ELECTROCHEMICAL STUDY OF HEAT TREATED SS304 STAINLESS STEEL IN SIMULATED BODY FLUID ENVIRONMENT

NIK MOHD NAZAIREN BIN NIK COB

Thesis submitted in fulfilment of the requirement
for the award of the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering
UNIVERSITI MALAYSIA PAHANG

DECEMBER 2010

UNIVERSITI MALAYSIA PAHANG

FACULTY OF MECHANICAL ENGINEERING

We certify that the project entitled "*Electrochemical Study of Heat Treated SS304 Stainless Steel in Simulated Body Fluid Environment.* "is written by *Nik Mohd Nazairen Bin Nik Cob*. We have examined the final copy of this project and in our opinion; it is fully adequate in terms of scope and quality for the award of the degree of Bachelor of Engineering. We herewith recommend that it be accepted in partial fulfilment of the requirements for the degree of Bachelor of Mechanical Engineering.

Luqman Hakim Bin Ahmad Shah

Examiner Signature

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature	....
Name of Supervisor:	PN JULIAWATI BINTI ALIAS
Position:	LECTURER OF MECHANICAL ENGINEERING
Date:	6 DECEMBER 20010

STUDENT'S DECLARATION

I hereby declare that the work in this project is my own except for quotations and summaries which have been duly acknowledged. The project has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature	....
Name:	NIK MOHD NAZAIREN BIN NIK COB
ID Number:	MA07058
Date:	6 DECEMBER 2009

ACKNOWLEDGEMENTS

I am grateful and would like to express my sincere gratitude to my supervisor Pn Juliawati Binti Alias for her germinal ideas, invaluable guidance, continuous encouragement and constant support in making this research possible. She has always impressed me with her outstanding professional conduct, her strong conviction for science, and her belief that a Degree program is only a start of a life-long learning experience. I appreciate her consistent support from the first day I applied to graduate program to these concluding moments.

My sincere thanks go to all my lab mates and members of the staff of the Mechanical Engineering Department, UMP, who helped me in many ways and made my stay at UMP pleasant and unforgettable. Many special thanks go to instructor engineer and assistance instructor for their excellent co-operation, inspirations and supports during this study.

I acknowledge my sincere indebtedness and gratitude to my parents for their love, dream and sacrifice throughout my life. I cannot find the appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals. Special thanks should be given to my committee members. I would like to acknowledge their comments and suggestions, which was crucial for the successful completion of this study.

ABSTRACT

This thesis is entitled about the electrochemical study of heat treated SS304 stainless steel in simulated body fluid environment. Metallic materials are generally used for biomedical devices for various parts of the human body. When a metal device is implanted into human body, it is continually exposed to extracellular tissue fluid. The exposed metal surface of the implant undergoes electrochemical dissolution of material at a finite rate due to the interaction with body environment. This interaction can lead to either failure of the implant or have an adverse effect on the patient resulting in the rejection of the implant by the surrounding tissue or both. In conjunction with that, it is important to improve the corrosion resistance and wear properties by conducting the heat treatment processes. The objectives of this study are, to study the corrosion behaviour of the heat treated SS304 stainless steel in phosphate buffered saline (PBS) and hank's balanced salt solution (HBSS) as simulated body environment and also to investigate the effect of heat treatment process to the corrosion rate of SS304 stainless steel in PBS and HBSS as simulated body environment. Heat treatment process, which is carburizing has been performed with different temperature and time. Carburizing is a process of adding Carbon to the surface. This is done by exposing the part to a carbon rich atmosphere at an elevated temperature and allows diffusion to transfer the carbon atoms into steel. The electrochemical study was conducted using a potentiostat WPG1000, interfaced to a computer in PBS and HBSS used for simulating human body fluid conditions. SS304 stainless steel show increased in corrosion rate after the carburizing process in simulated body fluid due to the resultant formation of chromium carbides.

ABSTRAK

Tesis ini membentangkan tentang kajian elektrokimia terhadap keluli tahan karat 304 yang terawat haba di dalam larutan berair (elektrolit) yang digunakan untuk mensimulasikan cecair tubuh manusia**.** Bahan-bahan logam biasanya digunakan untuk peranti bioperubatan untuk pelbagai bahagian badan manusia. Peranti logam yang ditanamkan ke tubuh manusia, adalah terus-menerus terdedah cecair rangkaian ekstraseluler. Permukaan logam implan yang terdedah mengalami kakisan elektrokimia pada peringkat terhad kerana interaksi dengan pesekitarannya. Interaksi ini boleh menyebabkan kegagalan implan atau mempunyai kesan buruk pada pesakit yang mengakibatkan penolakan implan dengan rangkaian sekitarnya atau kedua-duanya. Rawatan haba adalah penting untuk meningkatkan ketahanan terhadap pengaratan dan penghausan.Tujuan kajian ini adalah, untuk mempelajari perilaku pengaratan selepas rawatan haba besi tahan karat 304 di dalam cecair phosphate buffered saline(PBS) dan cecair hank's(HBSS) sebagai persekitaran badan simulasi dan juga untuk menyiasat pengaruh dari rawatan haba terhadap pengaratan besi tahan karat 304 di dalam PBS dan HBSS sebagai simulasi badan persekitaran. Rawatan haba yang telah dilakukan adalah karburasi dengan suhu yang berbeza dan masa yang berbeza. Karburasi adalah proses penambahan karbon ke permukaan sesuatu bahan. Hal ini dilakukan dengan mendedahkan sesuatu bahagian pada karbon yang tinggi pada suhu tinggi dan membenarkan resapan atom karbon ke dalam bahan tersebut. Penyelidikan elektrokimia yang dijalankan menggunakan WPG100 potensiostat, dihubungkan ke komputer dalam larutan berair (elektrolit) yang digunakan untuk mensimulasikan cecair tubuh manusia. Keluli tahan karat 304 menunjukkan peningkatan kadar pengaratan selepas proses karburasi di dalam simulasi cecair tubuh manusia disebabkan oleh pembentukan karbida kromium

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

4.5 Microstructure of SS304 stainless steel after carburizing for 8 hours at 950°C 41

4.6 Potentiodynamic polarization curves and Tafel extrapolation plot obtained in PBS at pH 7.4 and 37±2°C for SS304 Stainless Steel (carburizing for 8 hours at 950°C) in contact 45

LIST OF SYMBOLS

β_a	Anodic Slopes
β_c	Cathodic Slopes
$BaCO_3$	Barium Carbonate
$CaCl_2$	Calcium Chloride
CO_2	Carbon Dioxide
CO	Carbon Monoxide
Cr	Chromium
E_{corr}	Corrosion Potential
E_p	Primary Passivation Potential
Fe	Ferum
HCl	Hydrochloric acid
HNO_3	Nitric Acid
I_c	Critical Current Density
I_p	Passive Current Density
LogI	Log Current
KCl	Kalium Chloride
KH_2PO_4	Potassium dihydrogen
$MgSO_4$	Magnesium Sulphate
NaCl	Natrium Chloride
Na_2HPO_4	Disodium phosphate
$NaHCO_3$	Sodium Bicarbonate

LIST OF ABBREVIATIONS

ASTM	American Standard Testing Method
Ca	Calcium
CE	Counter electrode
CoCrMo	Cobalt-chromium-molybdenum
FYP	Final year project
HBSS	Hank's balanced salt solution
PBS	Phosphate buffered saline
RE	Reference electrode
RP	Corrosion resistance
SCC	Stress corrosion cracking
WE	Working electrode

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Nowadays, foreign materials have been routinely implanted in human body. The majority of these materials are made from metal alloys, such as stainless steels, cobalt chromium based alloys and titanium alloys. Major application of these materials are used as dental implants, orthopedic fracture plates, spinal rods, cranial plates, joint replacement prostheses and others. As the global population increases in age, the application of implants has increased rapidly.

The human body is not an environment that one would consider hospitable for an implanted metal alloys. Body fluid consists of aerated solution containing approximately 1% sodium chloride, together with minor amounts of other salts and organic compounds such as platelets, fat globules and chemical substances at a pH of 7.4. While it is well known that chloride solutions are among the most aggressive and corrosive to metals and ionic composition, protein concentration in body complicate the biomedical corrosion. When a metal device is implanted into human body, it is continually exposed to extracellular tissue fluid. The exposed metal surface of the implant undergoes and electrochemical dissolution of material at a finite rate due to the interaction with surrounding environment. This interaction can lead to either failure of the implant or have an adverse effect on the patient resulting in the rejection of the implant by the surrounding tissue or both.

The fundamental requirement for choosing a metallic implant material is that it can be biocompatible, that is not exhibiting any toxicity to the surrounding biological system. Biocompatible materials play an important and a critical role in manufacturing a variety of prosthetic devices in a modern world. Prosthetic devices are artificial replacements that are used in a biological system, such as the human body in an effort to provide the function of the original part.

For any material to act as a biomaterial, it must satisfy two essential characteristics that is bio-functionality and biocompatibility. Bio-functionality is related to a set of properties which allow a device to perform a required function, while biocompatibility refers to the ability of the device to continue to perform that function, effectively and as long as necessary, in or on the body. The biocompatibility of a material is affected by many factors, one of which and the most important one, is its corrosion resistance in a highly aggressive, high chloride containing body fluid environment.

The material must withstand the body environment and not degrade to a point that it cannot function in the body as intended. Materials used in the human body must not be treated or used in a configuration that would degrade the corrosion behavior. The implantation of biomaterials into the human body allows it to increase the quality of life and should have biological and chemical stability to improve functions of the human body.

1.2 PROBLEM STATEMENT

Metallic materials are generally used for biomedical devices for various parts of the human body. The interaction between the surrounding physiological environment and the surface of the implant materials can lead to bio-implant corrosion such as Pitting and Galvanic Corrosion. It is important to improve the corrosion resistance and wear properties by conduct the heat treatments process.

1.3 OBJECTIVES OF STUDY

The objectives of this study are:

(i) To study corrosion behavior of the heat treated SS304 stainless steel in PBS and HBSS as simulated body environment.
(ii) To investigate the effect of heat treatment process to the corrosion rate of SS304 stainless steel in PBS and HBSS as simulated body environment.

1.4 SCOPES OF PROJECT

The scopes of this study include:

(i) Sample preparation for electrochemical test.
(ii) Heat treatment process (carburizing).
(iii) Electrochemical test using potentiodynamic polarization technique.
(iv) Surface analysis of specimen by using Inverted Microscope.
(v) Corrosion rate analysis using IVMan software.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The use of materials as part of surgical implant is not new. It has been reported that substitution of bone parts in the body has been done since the late 18^{th} to 19^{th} century. In this era, bronze or copper were the main materials utilized. The implants used were successful but they have the problem of copper ion poisoning effect in the body. There were no other suitable materials for implantation were studied other than the copper and bronze until the mid-nineteenth century (Williams, 1987).

By the mid-nineteenth century, the medical sciences have already progressed in serious attempts to repair body parts with foreign materials. Following the introduction of stainless steels and cobalt chromium alloys in 1930s, greater success was achieved in fracture fixation and the first joint replacement surgeries were performed.

Biomaterial can be defined as a nonviable material used in a medical device, intended to interact with biological systems (Williams, 1987). Metallic components are generally used for orthopedics applications, since high strength and corrosion resistance are needed for bone repair and replacement.

2.2 STAINLESS STEEL

Metals are used as biomaterials due to have high wear and corrosion resistance. The most popular metallic biomaterials such as stainless steel, cobalt base alloys such as cast CoCrMo and titanium alloys. The first stainless steel utilized for implant fabrication was the 18-8 (type 302 in modern classification) which is stronger and more resistance to corrosion than vanadium steel (first metal alloys developed).

Stainless steels, in particular type SS304, are the most familiar and most frequently used alloy in the stainless steel family. Table 2.1 shows the SS304 stainless steel composition.

These alloys considered for a wide variety of applications because of the following properties:

(i) Resistance to corrosion
(ii) Ease of fabrication
(iii) Excellent formability
(iv) Beauty of appearance
(v) Ease of cleaning
(vi) High strength with low weight

Table 2.1: SS304 stainless steel composition

Element	**Composition**
carbon	0.08 max.
manganese	2.00 max.
phosphorus	0.045 max.
sulfur	0.030 max.
silicon	0.75 max.
chromium	18.00-20.00
nickel	8.00-12.00
nitrogen	0.10 max.
iron	Balance

Source: Thomas (1991)

Stainless steel implants are used as temporary implants to help bone healing, as well as fixed implants such as for artificial joints. Typical temporary applications are cranial plates, orthopedic fracture plates, dental implants, spinal rods, joint replacement prostheses, stents, and catheters. Figure 2.1 shows the phase diagram of stainless steel ternary. The main composition is chromium and nickel.

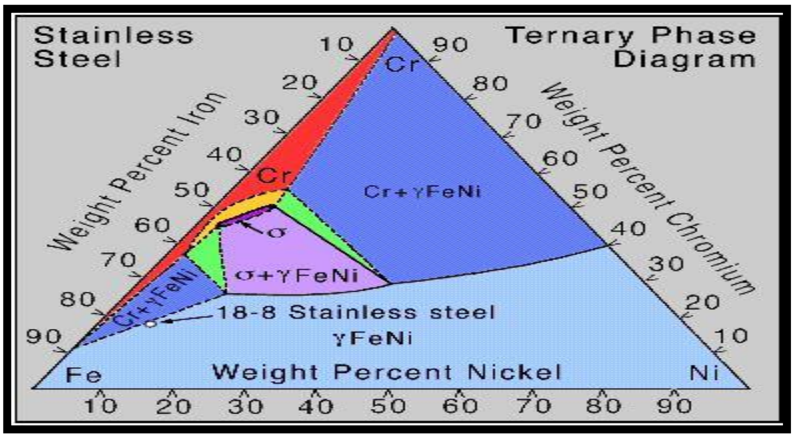

Figure 2.1: Phase diagram of stainless steel ternary.

Source: Adler (1991)

2.3 HEAT TREATMENT-CARBURIZING

Carburizing is a process of adding carbon to the surface. This is done by exposing the part to a carbon rich atmosphere at an elevated temperature and allows diffusion to transfer the carbon atoms into steel. This diffusion will work only if the steel has low carbon content, because diffusion works on the differential of concentration principle.

There are three well-known methods of carburizing. These methods introduce carbon by the use of solid compounds (pack carburizing), liquids (salt bath carburizing) and gas (atmospheric gas, plasma and vacuum carburizing). Figure 2.2 shows the case microstructure of stainless steel after air cooling from carburizing temperature.

Figure 2.2: Case microstructure of stainless steel after air cooling from carburizing temperature

Source: Iskander (1981)

2.4 CORROSION

Corrosion is defined as the destruction or deterioration of a material because of reaction with its environment. We have all seen corrosion and know that the process produces a new and less desirable material from the original metal and can result in a loss of function of the component or system.

The iron atom can lose some electrons and become a positively charged ion. This allows it to bond to other groups of atoms that are negatively charged. Oxygen, in the presence of water, accepts electrons to form hydroxyl ions. Oxygen dissolves quite readily in water and because there is usually an excess of it, reacts with the iron hydroxide.

2.41 Passivity

Passivity is refers to the loss of chemical reactivity experienced by certain metals and alloys under particular conditions. It occurs when the spontaneous formation of a hard non-reactive surface film that prevent further corrosion. This layer is usually an oxide or nitride that is a few atoms thick. Table 2.2 shows the passivity region. Passivity can be conveniently divided into three regions: active, passive, and transpassive.

Table 2.2: Passivity region

Regions	Behavior
Active	Identical as normal metal.
Passive	Slight increases in the oxidizing power of the solution cause a corresponding rapid increase in the corrosion rate. If more oxidizing agent is added, the corrosion rate shows a sudden decrease.
Transpassive	Very high concentrations of oxidizers or in the presence of very powerful oxidizers, corrosion rate again increased.

Source: Fontana (1986)

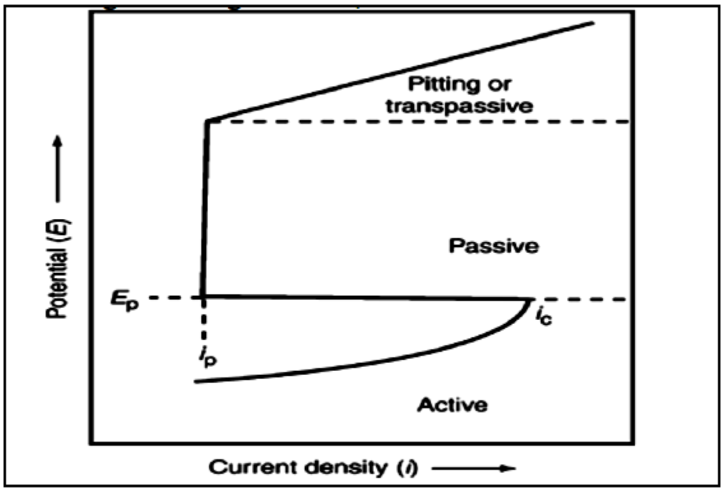

Figure 2.3: Passivity diagram

Source: Thomas (1991)